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FURTHER DEVELOPMENT, TESTING, AND APPLICATION TO COALS AND CLAYS USING A CONICAL FLUIDIZED BED

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Final Report

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Introduction

The working instrument was delivered, as agreed, on May 1, 1987. Assembly and acceptance testing of the system and computer software were finished and the results deemed satisfactory by May 15, 1987.

The studies completed at St. Francis Xavier University yielded several diskettes full of data, and the software programs necessary to display and manipulate these data were delivered along with the instrument.

The work at St. Francis Xavier and the preliminary testing at the Coal Research Center in Devon, Alberta indicate that the instrument is capable of yielding results reproducible within 3% when operated with identical charges under identical conditions. This reproducibility is deemed satisfactory but can probably be improved. The preliminary results show that a severe problem exists in the aging behaviour of most samples. Indeed even the ballotini spheres used to study the fluidized bed behaviour slowly react with the water and their properties do not stabilize for about three days. The problem of aging, especially with regard to coal samples, will be further discussed.

One problem does however remain. The pressure reading is very sensitive to the flow fields next to the probes. Thus the pressure reading not only reflects the static pressure due to the liquid plus suspended solid (i.e. slurry) density, but is also biased by the changing dynamics of the flow field. The flow field is not truly random, but rather contains a net upward component; thus, while averaging yields a reproducible value, it is not one containing the hydrostatic pressure alone. This problem will also be further addressed in the main body.

System as delivered.

1)-Hardware features.

The discussion and presentation of design elements contained in the previous reports will not be repeated. Only the improvements and modifications which were undertaken under the present contract will be discussed. These features can conveniently be grouped into "automation of probe positioning" and "modifications to the sampling probes to improve reliability under automatic operation".

1.a)-Automatic probe positioning.

The probe positioning sub-assembly was automated by using a stepping motor system configured for the Apple IIe by Rogers Labs. The system consists of a program card, resident within the computer, an external control and power supply system which operate a 400 steps per revolution high torque stepping motor. The external control system is housed in a chassis which also contains the power supply. Both of these items were manufactured at St. Francis Xavier's workshops.

Because of the probe's inertia, the size and power dissipation of the components is much larger than commonly found and generous air cooling had to be provided. The present configuration has worked for over four hundred hours in a lab enviroment without undue overheating.

The linear slide (Unislide^{tm}) support has a bearing screw of 1 mm pitch and hence positioning is precise to about .01 mm. A built-in Vernier gauge assures an accuracy and reproducibility of about .1 mm.

Choice of probe location is limited by the 400 mm length of the "Unislide^{tm}" platen. The operational area is, however, reduced to about 300 mm by the characteristics of the fluidized bed cone.

Serious damage can be done to the probe system if it is driven against the bottom (inlet) filter of the fluidized bed container. The only stops provided to guard against this eventuality are software stops. It is thus imperative that the initial probe position be properly set on startup and that the request for the current position of the probe not be defeated or incorrectly answered.

The alignment of the probes with respect to the vertical gantry and the vertical axis of the fluidized bed cone is critical. The parallelism of the cone axis to the gantry vertical motion axis can be adjusted by the three screw feet on the cone's tripod. The verticality or the gantry motion axis must be ensured during assembly by suitable manipulation of the levelling bolts on the support platform and by insuring that the gantry is securely bolted flush to the support platform. Further degrees of freedom in setting the verticality of the probes' motion are conferred by the fore and aft tilt which is controlled by a bolt at the base of the probe support and by a left-to-right tilt adjustment consisting of shims between the probe support and the gantry. Once set, all of these adjustments should be permanent. Deviation from verticality of the cone axis will lead to a badly conditioned fluidized bed, while deviation in the parallelism of the cone and gantry motion axis will lead to early failure of the probe.

1.b)-Sampling probes.

The glass probes used for manual operation proved to be too weak for reliable operation under computer control. Furthermore, the polyethylene sleeves used to maintain a sealed enviroment in the cone proved to bing excessively, leading to further stress and failure. To remedy this situation, a number of approaches were tried.

Metal tubes (e.g stainless steel, nickel etc.) proved to be strong enough but unsuitable because the e.m.f generated by contact with water completely swamped the desired signal. Several attempts were made at platinizing the metal tubes. Platinization, however, proved to be very difficult when a pinhole-free surface is needed. Further, even when successful, the lifetime of the platinized probes is short - about ten working hours.

Electropolishing of the tubes and preconditioning by deposition of a copper or nickel layer before the platinum layer was also tried. A pinhole-free coating can be reliably obtained in this way. The short lifetime of ten hours is however unchanged. Thus, the attempt to use a metallic tube was abandoned.

Careful observation of the failure modes of the sample probes revealed that the main stress was due to the sliding seal between the top plate of the conical fluidized bed vessel and the probe. Addition of vaseline increased the probe lifetime to about fifty working hours. Unfortunately vaseline releases surfactant-like materials into the circulating water and drastically effects results. Thus another way to reduce friction had to be found. The use of any liquid lubricating agent was rejected out of hand since it would lead to exactly the same problems as vaseline.

An effective solution was a teflon sheeting tube around a glass electrode. In the final configuration, the glass probes extend about five centimeters past the teflon sheaths. The teflon-to-glass unions are made waterproof by the use of heat-shrink tubes. This assembly has proven fairly reliable. This reliability can be ascribed to some amount of flexibility and to the fact that some breaking of the inner glass probe can be tolerated because the teflon sheath forms an airtight seal to the glass both at the

bottom (where the heat-shrink tubing is) and at the top where an O-ring seals the assembly into the sensor unit.

The teflon sheath or tubing alone does not make a suitable probe material because of excessive flexibility. The position of the probe ends (i.e. the measurement point) is very uncertain and not reproducible when the probe is moved if the probes are not fairly rigid. The diameter of the teflon tubing needed to obtain rigidity is too big for the present purpose and would introduce massive changes in flow pattern and velocity, thereby affecting measurements in a major and unpredictable way.

The torque needed to move the probes is large, requiring large current pulses to the stepping motor. Careful shielding of the cables and grounding of the shield to the gantry minimizes but does not completely smooth out the effect of these pulses on the high input impedance amplifier riding on the probe platform. Careful filtering and software design is necessary to achieve satisfactory performance. In particular, half-stepping the stepping motor at a relatively high frequency reduces the noise and takes it out of the response region of the amplifier, thus filtering out many of the unwanted effects. Averaging and proper timing of the sampling and A/D sequence also assist. These latter points will be further discussed in the software section.

2)-Software features.

2.a) Overview.

The software requirements for the system are predicated on a series of computer, control system, and measurement control requirements. The solution is an easily modified and very flexible system written in **Basic**. Computer performance is still adequate but will soon be overwhelmed if data

manipulation is increased. If computer performance is overwhelmed in the future, two solutions exist which do not require upgrading of the computer, with attendant reprogramming and purchase of new A/D and control interfaces. The cheapest is the purchase of a compiler for the system; this should speed up execution by a factor of ten to thirty and make the computer capable of much faster data manipulation speeds.

Reprogramming in Pascal would probably further increase execution speed and thus make data manipulation much easier. This would however involve a large investment in programming time and probably not be cost effective. Some system flexibility would also probably be lost because of the much more restrictive nature of Pascal programming.

2.b) Data acquisition and control requirement.

The data acquisition system used during manual operation, an already highly modified commercially available package, did not have the flexibility needed to handle the control and data filtering functions necessary for automated operation. Further information on this earlier system is available in previous reports. Also available is a sample video showing an example of operation and screen output.

In order to maintain data compatibility and some very efficient data manipulation, storage, and display features intrinsic to the commercial system, a decision was made to maintain data compatibility at the disk file level. Thus the requirement list finally decided upon was:

-Disk-file level compatibility with the older, manual system.

-Real time digital averaging.

-Up to ten A/D channels randomly enabled.

-Two channels of data displayed in real time as a graph.

-Channel of data displayed to be chosen at program start up.

-Full control of stepping motor to establish:

-a) Position at beginning of run.

-b) Mode of acquisition, i.e. while probe is being displaced (continuous or step-and-repeat).

-c) Motor speed and length of data acquisition pause.

-d) Direction of motion.

-e) Number of different positional points sampled.

-Full control of data acquisition interface to establish:

-a) Sampling rate.

-b) Input amplification factor.

-c) D/A output if necessary.

-d) Wait timing to ensure steady state data streams.

-Capability for some real time data averaging and manipulation.

-Hard copy of the data streams and/or the screen graphics.

2.c) Data acquisition and control software as delivered.

The company that supplied the data acquisition hardware system has available a software aid ("Adalab Toolkit tm") which allows full control of the A/D and D/A functions as well as channel selection and gain setting on individual channels. Since these are presented as machine language subroutines callable from a **Basic** program they can be integrated with any other **Basic** display and control program(s).

Rogers Labs, the supplier of the stepping motor system, also has available software to program the motor in **Basic**. Unfortunately, both the software and hardware were found to be very badly documented. Thus, even though final performance of the hardware when installed was satisfactory, installation and programming of the system was very difficult and time-consuming. Installation was successfully completed and a brand new software

driver for the stepping motor system written. This driver is much simpler and uses up far less memory than the one provided, while still yielding satisfactory performance. A manual with enough information to successfully operate the software has been subsequently written. Since this manual includes a full listing and operational procedures, these will not be duplicated here. A brief description of the program is however in order.

The main program first enables any or all of the five data channels now connected. These channels are:

Channel 0 - Total potential (fluidized bed inlet to top probe).

Channel 1 - Differential potential between probes.

Channel 2 - Pressure differential between probes.

Channel 3 - Conductivity of circulated water.

Channel 4 - Temperature or pH (controlled by hardware setting).

The data acquisition parameters and the two channels to be graphically displayed are also set by the operator under the guidance of the computer.

The program then requests the very critical data setting the present position of the probes. Provisions for probe positioning are also made in this section of the main program.

The motor control sequence is then determined. Parameters such as length of step, timing of data acquisition, number of steps and direction of steps are thus set under operator guidance. Software checks insure that the chosen sequence will not force the probes to exceed the allowable limits of travel. Since the effectiveness of these checks depends on the position of the probes entered into the program, accuracy of input is essential if hardware damage is to be avoided.

A series of subroutine calls then transfers control sequentially to motor stepping, timing, data acquisition and data manipulation modules.

This architecture allows for easy modification and editing of the program, since the parts likely to be modified reside in separate subroutines which are logically independent of the critical functions enabled in the main program.

Several data manipulation and presentation programs are then available to further manipulate the data and print it, in graphic form if necessary. In particular, several types of averaging routine have been developed. These routines have been of some help in analyzing system reproducibility and overall performance and copies have been provided on a program diskette. To ensure a good degree of functionality a common data file format is used in all of these programs.

Results

1) Delivered System.

As shown by the data contained in diskettes "Cone Data vol 1 to 13" which were acquired at St. Francis Xavier and the six diskettes of data acquired at Devon during commissioning, the system is in working order. It will acquire and display data on any or all of the five available data input channels and control probe positioning to within 0.1 mm, the Vernier scale resolution. Thus the hardware and software parts of the instrument meet all mechanical and electronic requirements. Reliability of the new probes appears to be good if reasonable care is exercised. Further, these probes can easily be rebuilt with the materials provided; enough material to construct an extra six probes (three pairs) is available.

2) Data quality.

Initial tests of data quality were disappointing. Upon comparison of subsequent data acquisition runs with the same sample, a persistent and

severe drift was detected in both the differential and total developed potential measurements on both coal and ballotini samples. Further testing indicated that the drift diminished with the amount of time the sample was in contact with the water.

A series of runs over a period of a week then clearly showed that the drift was not an instrumental factor, but rather was due to the slow kinetics of the reaction, and hence the slow equilibration, of the sample with water. The slow drift in potential was also reflected in changes in the pH and the conductivity of the water over the contact period. Further experimentation revealed that the ballotini and coal need at least four and three days, respectively, to equilibrate with the water enough so that two experimental runs undertaken one hour apart do not differ by more than the random errors intrinsic in each run. Consequently, the aging should be done in a separate vessel in order to optimize the use of the instrument.

Since the instrument is now known to be reliable, and in view of the typically very short slurry conditioning times available in most industrial applications, a protocol can be instituted calling for measurement to begin at some constant, but arbitrary, time (e.g. half an hour) after the wetting of the sample. If an appropriate aging time is chosen and then used consistently, the results should reflect trends in surface characteristics which parallel plant experience.

The problem is however more serious if studies of surface active ingredients are to be undertaken. Since the system should be unchanged except for the effects of the surfactants, the full three-day aging must be performed if the surfactant contribution is not to be masked by the surface hydrolyzation or oxidation reactions which are presumably responsible for the aging process.

When operated with well-aged samples, it is possible to conclude that the system has a reproducibility of about 2%. This figure is dependent on such parameters as averaging time and mode as well as data acquisition speed, but is nevertheless typical of the results obtained using the DATACQ.8 program available on the diskettes. Figures 13 and 14 a,b illustrate this reproducibility. Figure 13 shows two pressure run and their difference. The difference scale is magnified by a factor of four to make the deviation visible. The position of the arrow indicate probe turnaround point. Figure 14 a shows the total differential readinds for the same runs while 14 b is the superimposition of the two runs in 14a.

The reproducibility of the pressure readings above the fluidized bed level are an apparent exception to the above figure of 2%. This area is exemplified by the wavy region at the beginning and at the very end of the difference plot in figure 13. This uncertainty is not due to the data acquisition system, but rather to the slowly evolving and quasi-random nature of the fluid flow in this region. Being essentially random, the flow appears noisy; however, since this variation is relatively slow, it is impractical to acquire data for a long enough time to average these effect. Within the fluidized bed itself, the time constant for changes is much shorter so that a stable average can be obtained within a reasonable time interval (5-10 secs/position).

3) Interpretation of data.

The interpretation of the data according to equations available in the literature (Adorjan et al.) is not successful in our system. The stumbling block is a pressure difference term which is proportional only to the density of the bed and does not include the prevailing flow fields within

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Another researcher (J. Masliyah of the University of Alberta's Chemical Engineering Department comes to mind) should be comissioned to undertake a brief contract to model flow patterns and their associated pressure variations within the conical fluidized bed.

Notwithstanding this major problem, the apparatus has already proved its worth by elucidating various problems and observation encountered during its construction and testing; some of these were already discussed in the previous reports but will be briefly mentioned here.

The effect known as the "Donnan potential" has long been appreciated as a source of error when measuring pH in a particles (typically cells) laden slurry. The relationship to the charge of the particles had been inferred from the observation that highly charged cells (such as erythrocites, the red blood cells) give rise to a measurable effect even while settling under normal (1g) gravitational fields. However, no report was discovered which discussed the possibility that this effect might be several pH units in magnitude. The typical effects reported in the literature are in the range of .1 to .3 pH units. The effects which we measured, and reported previously, have a maximum of about 3 pH units. These observations have profound implications whenever the pH of a strongly stirred slurry of relatively large particles (over .3mm) has to be monitored for pH effects. The problem was solved by constructing a shielded probe in a platinized brass tube connected to ground. A more elegant solution would be to use fiber optic systems with indicators encapsulated at the end of the fibers. It is of some interest to note that the generated potentials (a few hundred millivolts) are large enough to lead to serious corrosion problems for most metals.

The theoretical expectation that, if a sufficiently large amount of solid is used, a layer of unfluidized material will be formed above the fluidized bed was confirmed experimentally. This observation has commercial implications, especially in the design and operation of sludge blanket systems for waste water (sewer) treatment.

The long time necessary to establish an equilibrium between water and the solid dispersed in it was also a surprise. While this aging effect is a well known one, previous reports seem to indicate that 12 to 24 hours is a sufficient time interval. The shorter times reported in the literature are perhaps related to the experimental errors associated with streaming potential and electrophoretic measurements. Because of packing problems in the case of streaming potential, and sampling errors in the case of electrophoretic measurements, the expected uncertainties in these measurements are around ten percent. Further, the measurement time is typically three to five minutes, compared to the half to one hour for the present system. Thus, in view of the better reproducibility and longer times required to repeat the measurements, the stability requirement in obtaining duplicate or triplicate readings are much less stringent in the traditional techniques of streaming potential and electrophoresis. These laxer constraints would naturally lead to apparently shorter aging times to experimental stability.

Clearly more research is necessary before the very desirable goal of measuring the zeta potential of particles in water under conditions of size, concentration and shear forces similar to those found in industrial processing environments can be fully realized. The present instrument may, however, be used to study relative surface electrical effects and therefore to validate or contradict models of surface characteristics. As mentioned

above, validation of these measurements with the more traditional electrophoretic and streaming potential methods has proven impossible because of the problems associated with the measurement of the relative velocity of the liquid and the solid. These data are usually assumed to be proportional to pressure differences in the streaming potential technique. As previously stated this assumption is not valid in this instrument.

Future work should concentrate on correlating the results from more traditional methods with fluidized cone measurements. This could perhaps be approached empirically by conducting a sufficiently large number of tests to allow development of a statistically correct empirical correlation. Since such a correlation would probably be non-linear, and since flow rate, particle density, and fluid composition and viscosity would all play a part, several tens of tests might, in fact, be necessary. A rather exhaustive analysis to derive the multi-dimensional response surface correlating the data would also be required. Further, being purely empirical, the envelope of reliability of this relationship would be largely unknown. Thus, this is certainly not an option to be recommended. Some numerical calculation on the expected flow and pressure to be expected should prove to be more rewarding.

Once the data handling problem is solved the equipment lends itself to a variety of projects. For example:

The design allows flotation experiments to be easily done in situ. Measuring electrical properties before and after flotation will allow more accurate studies of the effects of surface characteristics on flotation.

Titration against surfactants is possible and their effects on surfaces can thus be studied.

Thus when the data interpretation problems are overcome this equipment should prove to be extremely flexible and useful. Note that some of these research project can be in fact undertaken, and will yield useful results, now. The only possible drawback will be in the lack of correlation to previous literature. However, since previous literature on slurries of several or several tens of percent concentration is remarkably skimpy and unreliable, the lack need not be crippling.

Figure 1

Figure 2a

1=IN/OUT 2=UTILITY 3=NUMERIC 4=SPECIAL
1=IN/OUT 2=UTILITY 3=NUMERIC 4=SPECIAL

Figure 2b